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Calcination of Calcium Hydroxide Sorbent in the Presence of SO2 and Its Effect on Reactivity

Acurex Corp., Research Triangle Park, NC

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16. ABSTRACT The paper discusses the calcination of Ca(OH)2 sorbent in the presence of SO2 and its effect on reactivity. When Ca(OH)2 is calcined in an isothermal flow reactor with 300 ppm or less SO2, the structure of the sorbent is characterized by retention of higher pore volumes and surface areas than when calcined in the absence of SO2. When reinjected into the isothermal flow reactor (1000 C, 3000 ppm, 0.8 s), a higher level of reactivity with SO2 is observed for the SO2-modified calcine than for the unmodified sorbent. Structural evidence suggests that a slower initial rate of sintering in the modified calcine accounts for its enhanced reactivity. It is postulated that deposition of small amounts of CaSO4 on pore faces and along crystal boundaries retards the solid state diffusion process responsible for sintering. The effects on sintering and reactivity of various combinations of residence time (0.6 - 2.0 s) and SO2 concentration (10 - 50 ppm) during precalcination have been tested.

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Sorbents	Stationary Sources	11G
Calcium Hydroxides		07B
Sulfur Dioxide		
Calcines		11B
Reactivity		07D
Calcium Sulfates		

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CALCINATION OF CALCIUM HYDROXIDE SORBENT IN THE PRESENCE OF SO_2 AND ITS EFFECT ON REACTIVITY

by

Wojciech Jozewicz*
Acurex Corporation
P. O. Box 13109
Research Triangle Park, North Carolina 27709

and

David A. Kirchgessner
U. S. Environmental Protection Agency
Air and Energy Engineering Research Laboratory
Research Triangle Park, North Carolina 27711

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*Person to whom questions concerning this paper should be addressed

ABSTRACT

When calcium hydroxide [$Ca(OH)_2$] is calcined in an isothermal flow reactor with 300 ppm or less sulfur dioxide (SO_2) present, the structure of the sorbent is characterized by retention of higher pore volumes and surface areas than when calcined in the absence of SO_2. Upon reinjection into the isothermal flow reactor (1,000°C, 3000 ppm SO_2, 0.8 s) a higher level of reactivity with SO_2 is observed for the SO_2-modified calcine than for the unmodified sorbent. Structural evidence suggests that a slower initial rate of sintering in the modified calcine accounts for its enhanced reactivity. It is postulated that deposition of small amounts of calcium sulfate ($CaSO_4$) on pore faces and along crystal boundaries retards the solid state diffusion process responsible for sintering. The effects on sintering and reactivity of various combinations of residence time (0.6-2.0 s) and SO_2 concentration (10-50 ppm) during precalcination have been tested.

INTRODUCTION

The limestone injection multistage burner (LIMB) process developed by EPA is designed to control sulfur dioxide (SO_2) emissions from coal-fired boilers through the injection of dry calcium-based sorbents into the high temperature zone of the furnace (about 1,230°C). Research has shown that calcium hydroxide [$Ca(OH)_2$] is more reactive with SO_2 in this process than are other commercially available sorbents.[1,2,3,4] Once this fact had been established, considerable experimental effort was devoted to understanding the fundamental kinetic behavior of $Ca(OH)_2$ in high temperature injection processes, so that SO_2 capture could be predicted.

Calcination of both calcium carbonate ($CaCO_3$) and $Ca(OH)_2$ has been studied extensively at the laboratory scale, and has been modeled most recently by Silcox et al.,[5] and by Milne et al.[6,7] Milne used the data of Bortz and Flament[3] and Mai[8] to develop a rate constant appropriate for the calcination of $Ca(OH)_2$. Sintering of the resulting calcium oxide (CaO) is not as well understood and no theoretical model for it currently exists. Sintering is important because the loss of specific surface area and porosity through this solid state diffusion process competes

directly with the process of sulfation. An empirical sintering model has been proposed by Silcox et al.,[5] however, and has been shown to effectively correlate the data of Borgwardt et al.[9]

Of particular interest in the sintering process is the effect of combustion gas composition, since this is the environment of practical concern. Wide agreement has been reached on the catalytic effect of CO_2 on sintering of CaO. This effect has been documented by Anderson et al.,[10] Ulerich et al.,[11] Borgwardt,[12] Borgwardt and Bruce,[13] Borgwardt et al.,[14] and Mai,[8] and is incorporated in Silcox's rate equation. The catalytic effect of water on sintering has been shown by Mai[8] and Borgwardt,[12] and can also be included in the Silcox equation. Presently, the Silcox equation does not account for the effect of SO_2 on sintering.

There appears to be a discrepancy in the available data on the effect of SO_2 on sintering. Glasson[15] notes the ability of calcium sulfate ($CaSO_4$) product to promote sintering, and Newton[16] has suggested that SO_2 may promote sintering as well. Lange[17] observes, however, that a small amount of SO_2 in the calcination gas yields a CaO that is more reactive with SO_2 upon furnace injection than a CaO calcined in the absence of SO_2. A similar

effect is seen by Kirchgessner and Jozewicz.[18] Before the effect of SO_2 on sintering can be incorporated into the rate equation of Silcox, the effect must first be verified, and then quantified. The purpose of this paper is to verify the inhibition of sintering by SO_2 as noted by Kirchgessner and Jozewicz. A secondary purpose is to determine the practical application of precalcining sorbents in the presence of small amounts of SO_2 prior to furnace injection as suggested by Lange.[17]

EXPERIMENTAL SYSTEM

Hydration

The parent material for the $Ca(OH)_2$ used in this study is a commercial CaO produced by the Longview Lime Division of Dravo Lime Co., Saginaw, Alabama. Hydroxides are produced in a steam-jacketed Ross ribbon blender loaded with 6.8 kg pulverized CaO and allowed to reach a temperature of 30-50^{o}C. A total of 3.7 kg water is then added at a constant rate for 40 min. The blender is run for an additional 20 min while the heat of reaction drives off excess water, leaving a dry $Ca(OH)_2$ product.

Reactor

The electrically heated isothermal flow reactor into which the hydrates are injected for structural and sulfation studies is shown in Figure 1. The reaction chamber consists of two concentric, 3.35 m long quartz tubes heated by three Lindberg tube furnaces. The inner tube has a 15 mm ID, and the outer tube has a 50.8 mm OD. Premixed gases enter the reactor at the bottom of the annulus between the inner and outer tubes and are heated as they move upward. At the top of the reactor they enter the inner tube with the sorbent and move downward through the reaction zone. Gases and solids exit the reactor through an air-cooled heat exchanger that quenches the reactant stream to approximately 280°C. Solids are captured in a cyclone followed by a glass-fiber filter. Sorbent is fed from a fluidized bed feed tube through 1.9 mm ID syringe tubing. For this study the reactor is operated at nominal residence times of 0.6-2.0 s. Residence time is adjusted by varying gas flow, at standard temperature and pressure (25°C, 760 mm Hg [101 kPa]) from 25.54 to 5.86 L/min. Gas composition is 95 vol. % N_2, 5 vol. % O_2, and from 10 to 3000 ppm SO_2. Although they would be of interest, SO_2 concentrations below 10 ppm cannot

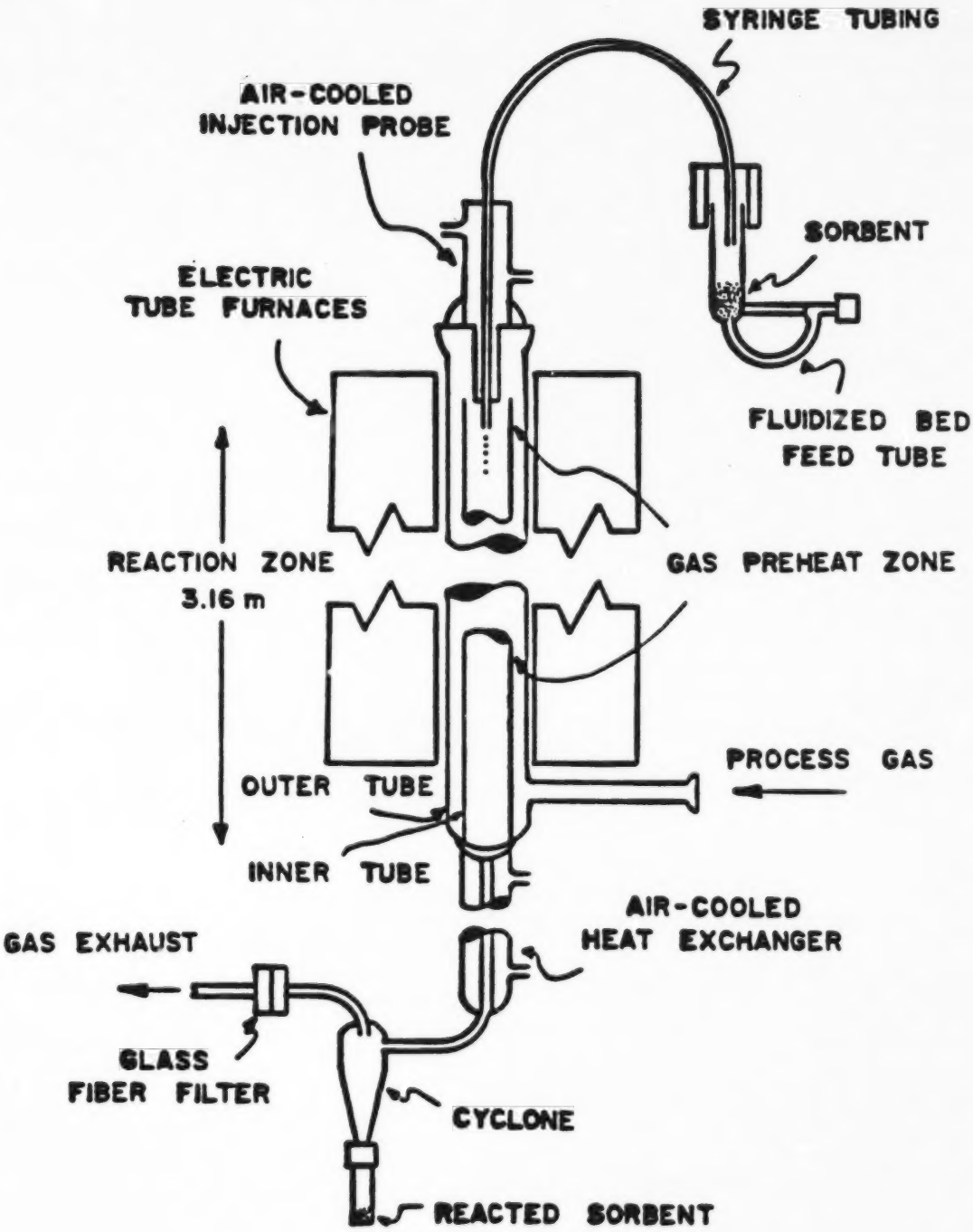

FIGURE 1. ISOTHERMAL FLOW REACTOR.

be well controlled. Due to variability in the reactor system, selected samples are replicated until standard deviation falls to less than 10% of the mean.

Analysis

Calcium utilization or conversion of reactor products is calculated after analyses for calcium by atomic absorption spectrophotometry, and for sulfate by ion chromatography. Calcium utilization analyses have a precision of ±5%. No sulfur species other than sulfate have been detected. Specific surface area and porosity are measured by N_2 adsorption/desorption in a Micromeritics Digisorb 2600 autoanalyzer. Machine error is less than ±1%.

RESULTS

In order to isolate the effect of SO_2 on sintering from the effects of time and temperature on sintering, $Ca(OH)_2$ was initially calcined in the presence of SO_2 concentrations varying from 10 to 5,000 ppm, while holding time and temperature constant at 0.8 s and 1,000^{o}C, respectively. The results are shown in Figure 2. In the absence of SO_2, $Ca(OH)_2$ will calcine to CaO

having a maximum surface area of about 76 m^2/g (Borgwardt,[19] Kirchgessner and Jozewicz),[20] and then sinter to successively lower surface areas. At 1,000°C and 0.8 s, the CaO has sintered to a surface area of 24 m^2/g. Since $CaSO_4$ has a volume approximately three times that of the CaO reactant, one would expect any degree of sulfation to immediately reduce surface area below 24 m^2/g through the processes of pore filling and pore occlusion. It can be seen from Figure 2, however, that calcination of $Ca(OH)_2$ in the presence of SO_2 concentrations of 10 - 300 ppm results in higher surface areas than calcination in the absence of SO_2. The calcium conversions accompanying these SO_2 concentrations are 3.4 - 11.0% and must cause fine pore filling and blocking. For surface areas greater than 24 m^2/g to occur, therefore, the resistance to sintering which accompanies these conversions must be significant. An SO_2 concentration of 10 ppm causes the greatest amount of surface area to be retained, with successively more surface area being lost at higher SO_2 concentrations and calcium conversions. Above 300 ppm SO_2, retained surface area falls below 24 m^2/g due to buildup of the $CaSO_4$ produce layer. Figure 3 shows the behavior of porosity under the same conditions as Figure 2. Porosity reduction mirrors the trend in surface area reduction throughout the range of SO_2 concentrations and calcium utilizations as expected.

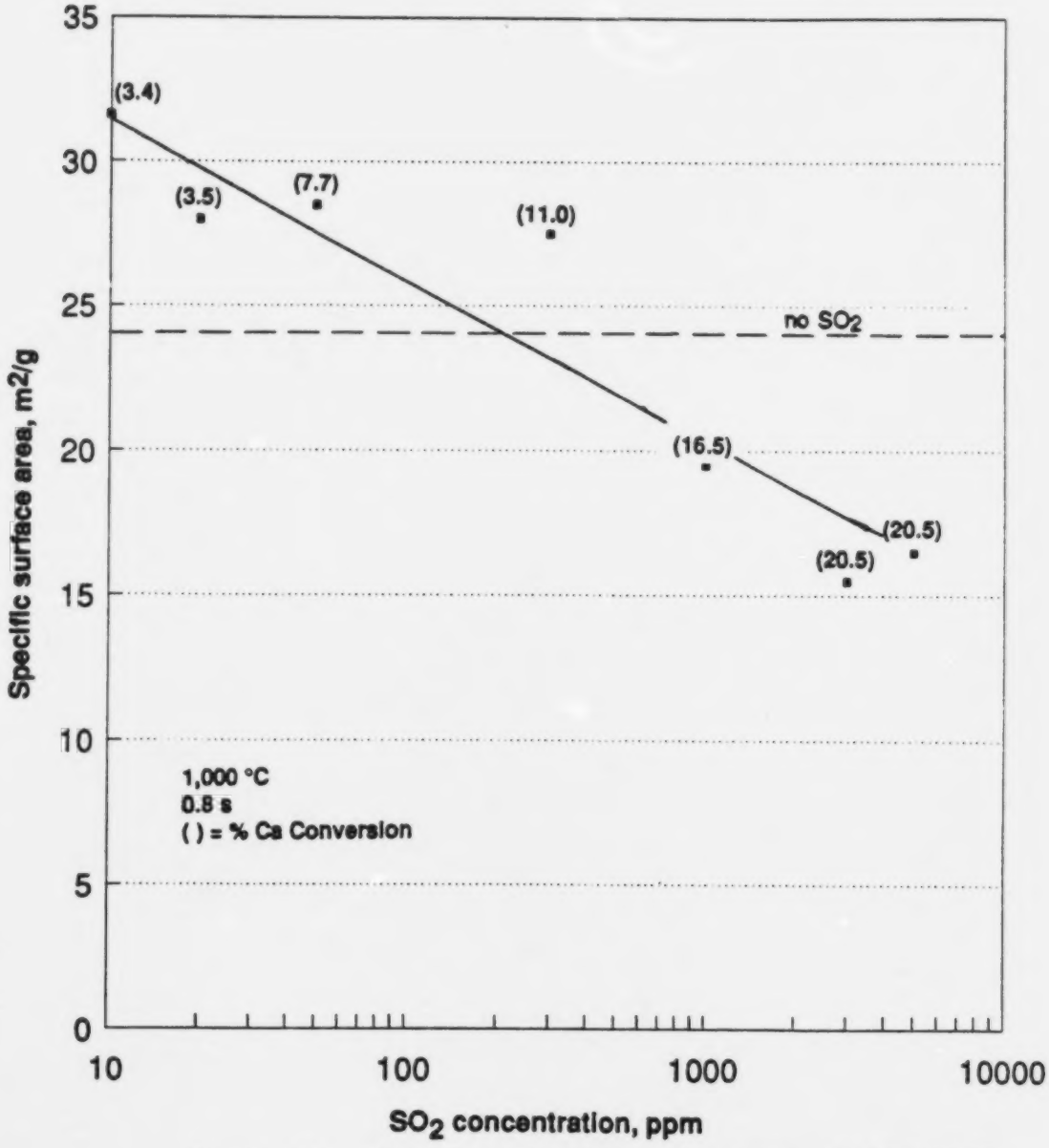

FIGURE 2. SURFACE AREA RETAINED AFTER CALCINATION WITH AND WITHOUT SO_2.

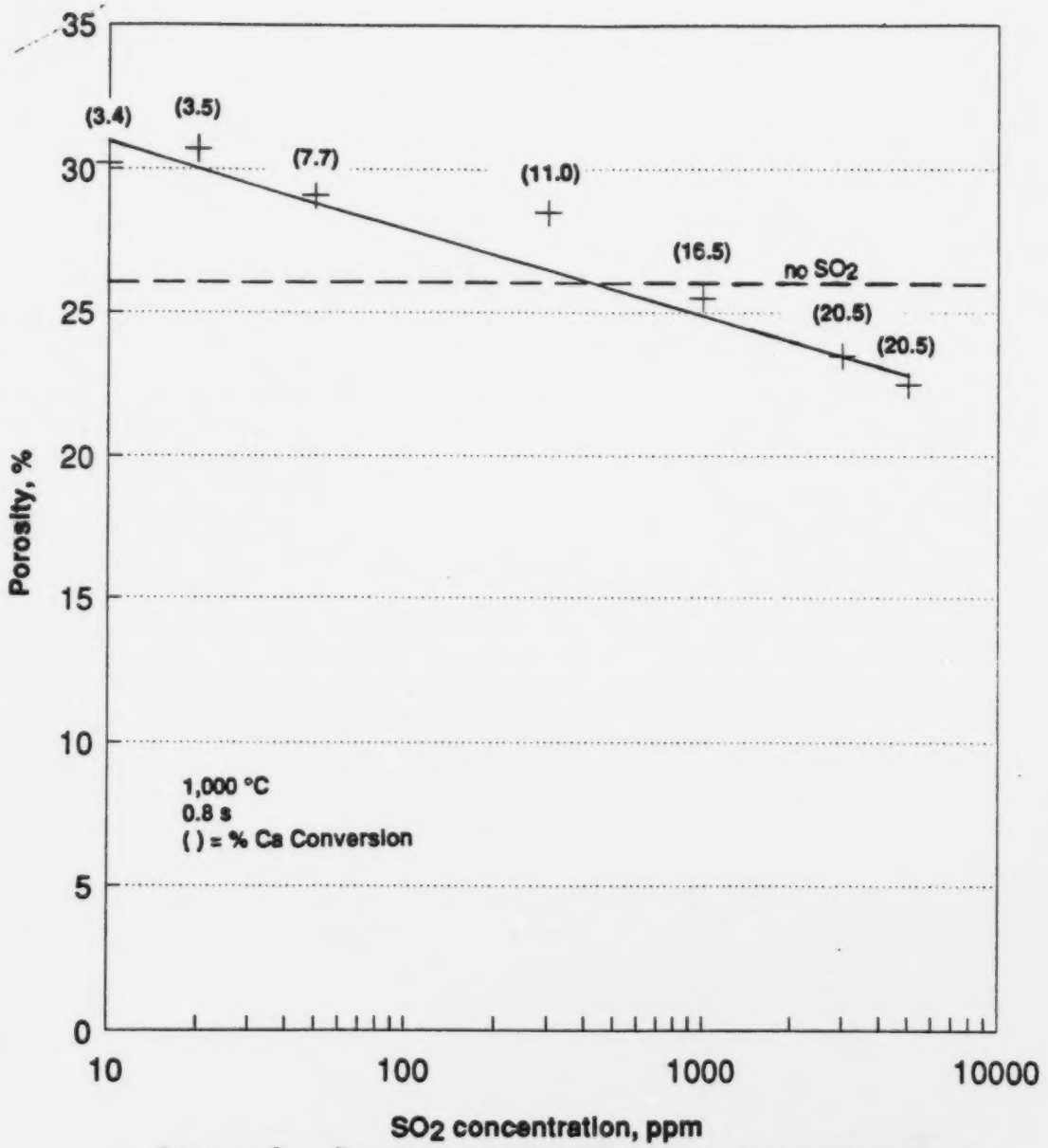

FIGURE 3. POROSITY RETAINED AFTER CALCINATION WITH AND WITHOUT SO_2.

To determine the effect on structure of calcination or contact time in the presence of SO_2, Figures 4 and 5 were produced. Figure 4 shows that, in the absence of SO_2, thermal sintering reduces surface area over time as one would expect. In the presence of 10 and 50 ppm SO_2, thermal sintering reduces surface area at approximately the same rate as in the absence of SO_2 over the 0.6-2.0 s time frame investigated. In both cases, however, the surface areas retained in the presence of SO_2 are higher than in the absence of SO_2. This implies that, during the 0 - 0.6 s time period, the rate of sintering in the absence of SO_2 is greater. At all points, the surface area retained in the presence of 10 ppm SO_2 is greater than that retained in the presence of 50 ppm SO_2. As in Figure 3, the porosity behavior in Figure 5 reflects the trends in the surface area data.

The loss of surface area through sintering is known to be a significant factor in reducing the reactivity of $Ca(OH)_2$ with SO_2 during furnace injection. Since the presence of small amounts of SO_2 during calcination appears to retard the initial rate of sintering, it has been suggested that this behavior may have a practical application in sorbent injection processes. Lange[17] suggests that precalcination of sorbents in the presence of

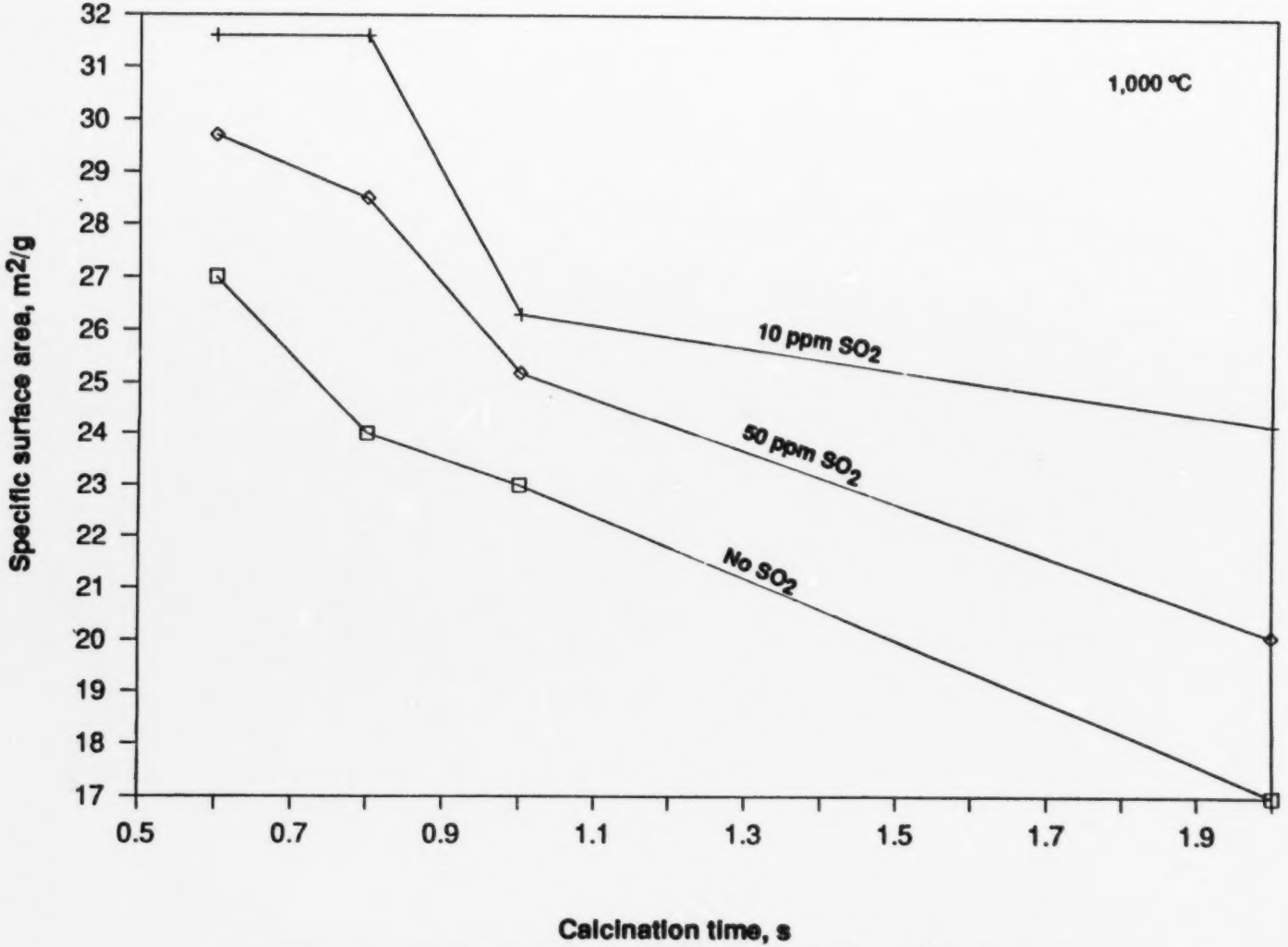

FIGURE 4. VARIATION IN SURFACE AREA WITH SO_2 LEVELS AND TIME DURING PRECALCINATION.

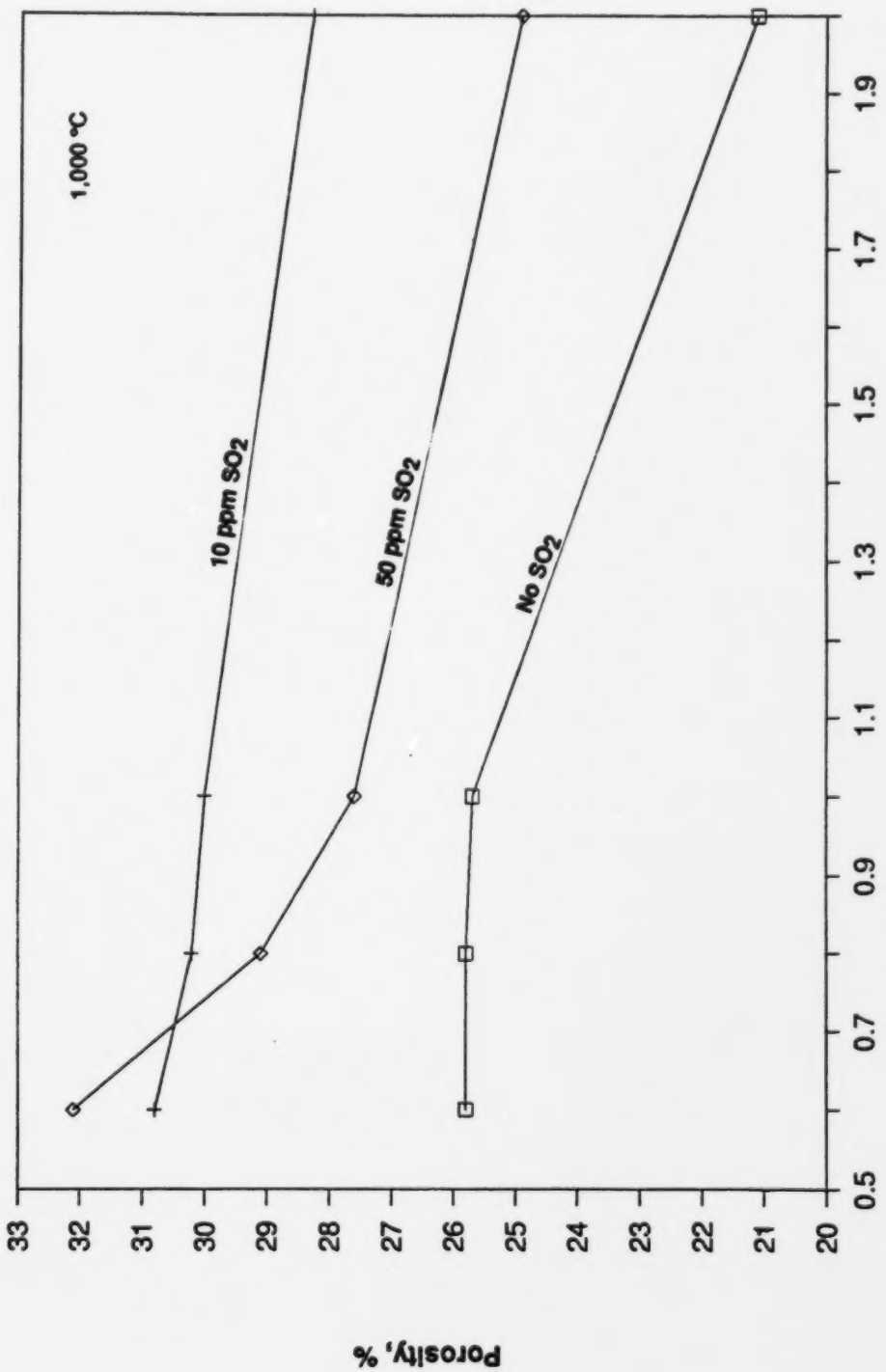

FIGURE 5. VARIATION IN POROSITY WITH SO_2 LEVELS AND TIME DURING PRECALCINATION.

small amounts of SO_2 will produce precalcines that are highly reactive with SO_2 during reinjection into a boiler. This would be of interest because high surface area precalcines have been shown to be less reactive with SO_2 during furnace injection than their uncalcined counterparts (Bortz et al.).[21]

To test this hypothesis, sorbents were precalcined with and without 10 ppm SO_2 for times of 0.6 - 2.0 s. These sorbents were then reinjected into the flow reactor at 1,000°C for 0.8 s with 3,000 ppm SO_2 to determine differences in calcium utilization. The results are shown in Figure 6. The lowermost curve reflects the level of calcium conversion achieved at various precalcination times in the presence of 10 ppm SO_2. The uppermost curve shows the levels of additional calcium conversions achieved when these same sorbents were reinjected at 1,000°C for 0.8 s with 3,000 ppm SO_2. These values are reported separately because they would reflect the only SO_2 capture of interest if the precalcined sorbents were to be used in a furnace injection process. The middle curve shows the calcium conversion obtained during reinjection by the sorbents precalcined in the absence of SO_2. It is clear that, for all precalcination times, sorbents precalcined in the presence of 10 ppm SO_2 produce higher levels of calcium

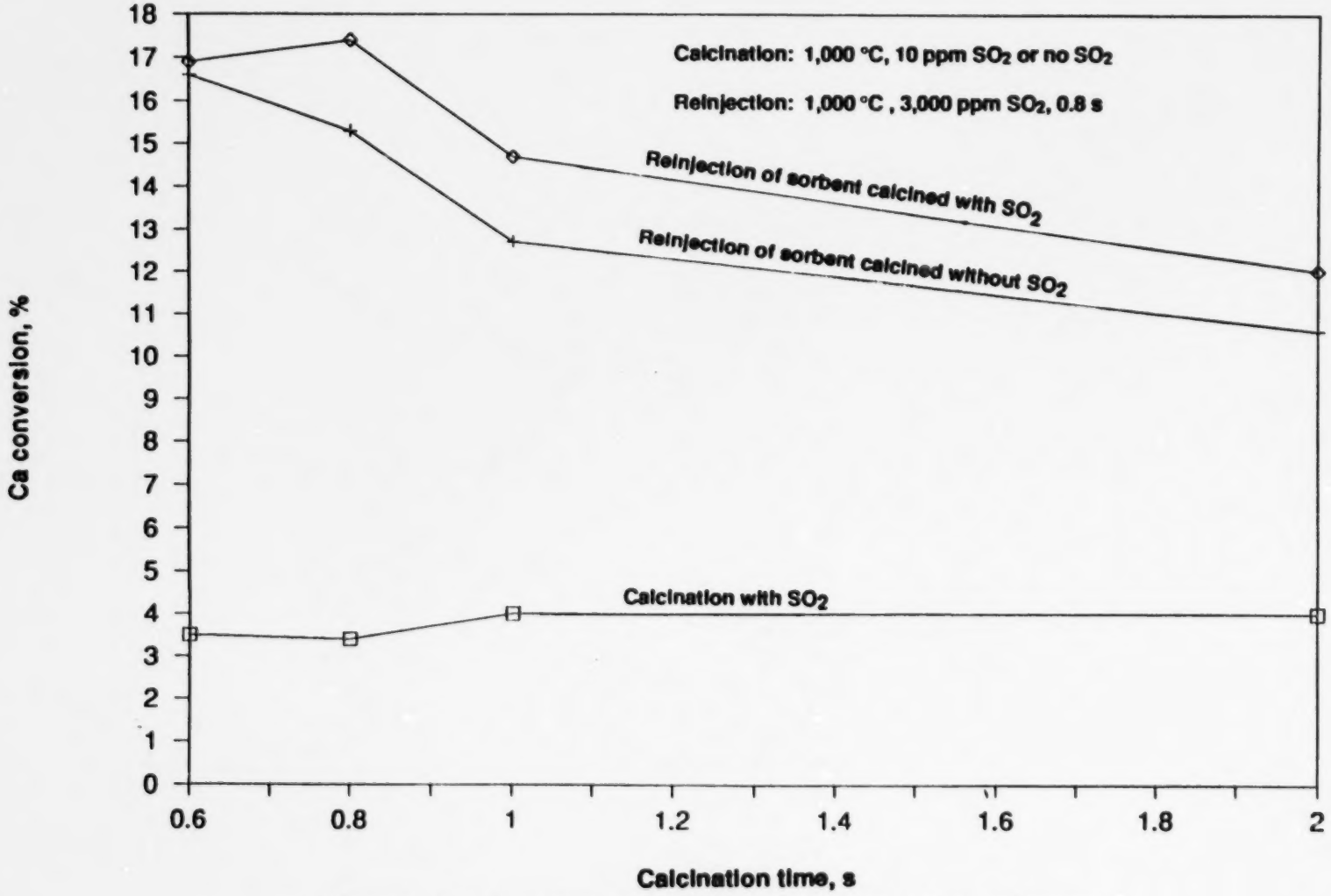

FIGURE 6. CALCIUM CONVERSION ACHIEVED AT A CONSTANT REINJECTION TIME BY SORBENTS PRECALCINED WITH AND WITHOUT 10 PPM SO_2.

conversion during reinjection than sorbents precalcined in the absence of SO_2.

Figure 7 shows the time-resolved reactivity during reinjection of sorbents precalcined with and without 10 ppm SO_2 for a constant time of 1.0 s. Again, the lowermost curve reflects the constant calcium conversion value achieved during precalcination in the presence of SO_2. The uppermost curve shows the calcium conversion obtained at various residence times during reinjection at 1,000°C with 3,000 ppm SO_2. These values are, again, consistently higher than calcium conversions demonstrated by sorbents precalcined in the absence of SO_2. The shape of the curve for SO_2-modified precalcined sorbents reinjected into the reactor suggests a significant influence of pore diffusion, while the curve for precalcined sorbents generated without SO_2 is indicative of produce layer diffusion. This would imply that sintering has not yet removed the fine pore structure in the SO_2-modified precalcined sorbents.

In Figure 8, the result of increasing the SO_2 available during precalcination to 50 ppm is shown. In this case sorbents are precalcined with and without 50 ppm SO_2 at 1,000°C for times of 0.6 - 2.0 s. As before, these sorbents are then reinjected

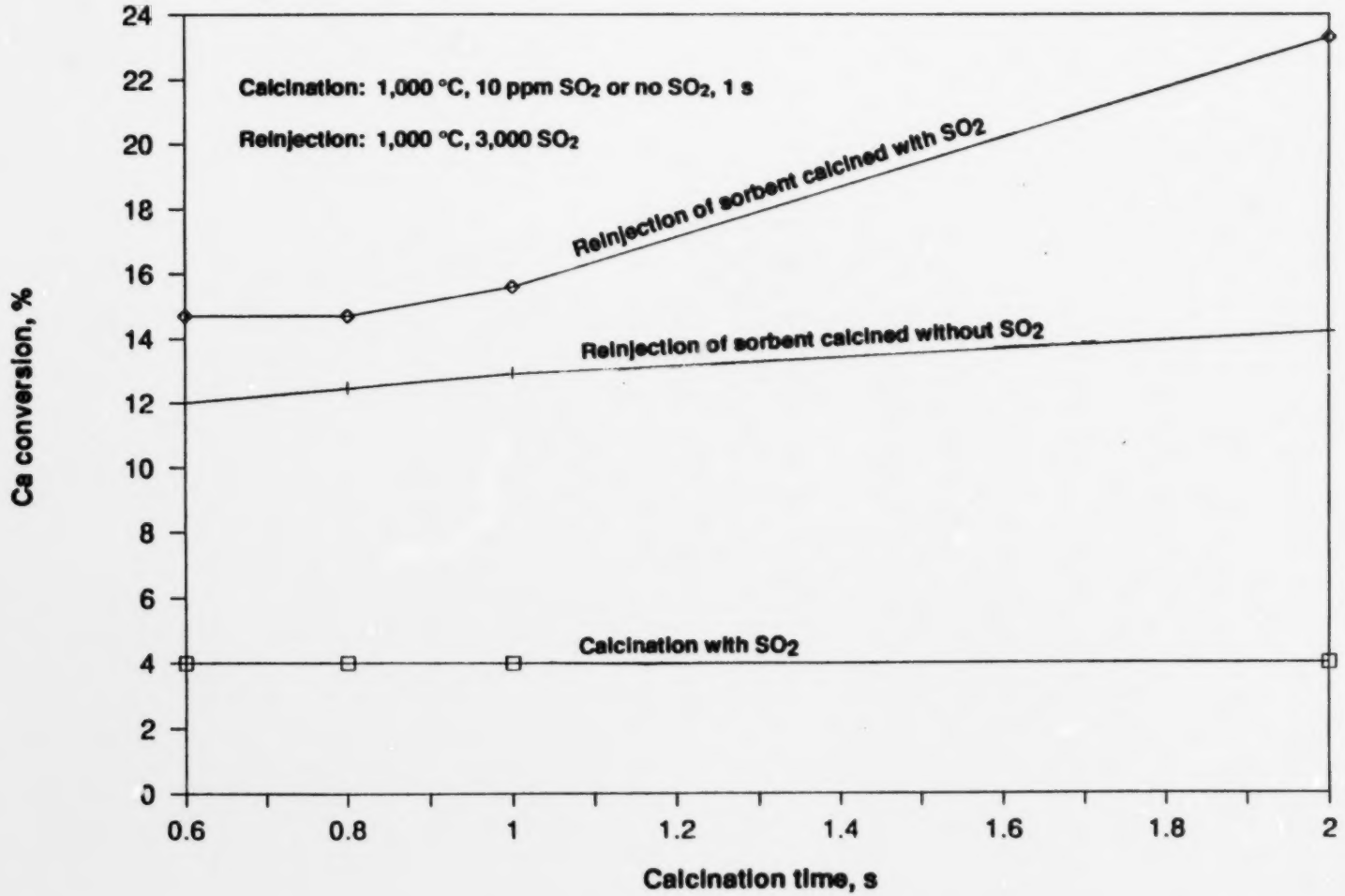

FIGURE 7. CALCIUM CONVERSION ACHIEVED DURING REINJECTION BY SORBENTS PRECALCINED AT A CONSTANT TIME WITH AND WITHOUT 10 PPM SO_2.

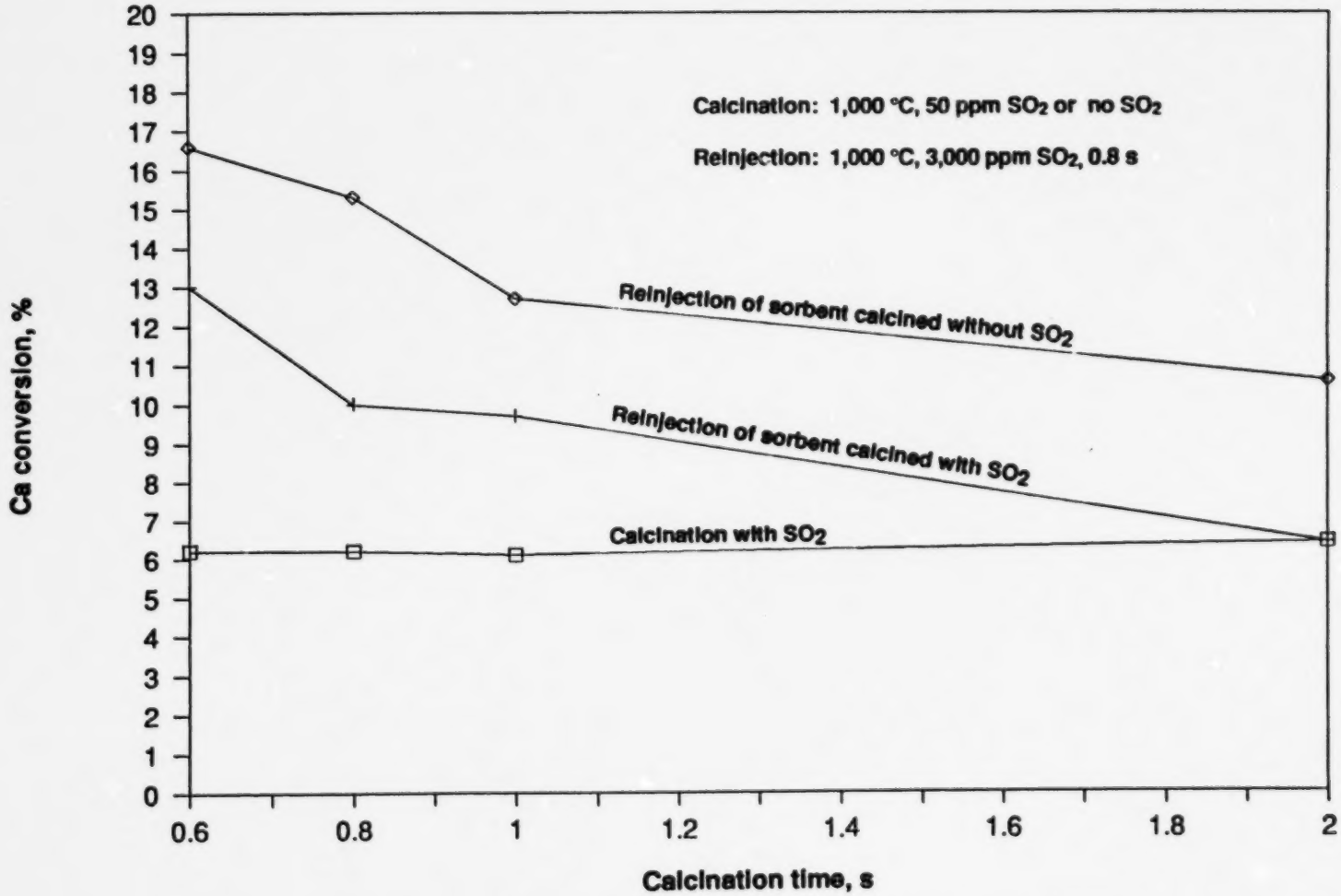

FIGURE 8. CALCIUM CONVERSION ACHIEVED AT A CONSTANT REINJECTION TIME BY SORBENTS PRECALCINED WITH AND WITHOUT 50 PPM SO_2.

into the flow reactor at 1,000°C for 0.8 s with 3,000 ppm SO_2 to determine differences in calcium conversion. In contrast to the sorbents modified with 10 ppm SO_2, sorbents modified with 50 ppm SO_2 are less reactive during reinjection than sorbents precalcined in the absence of SO_2. Figures 2-5 suggest that sorbents modified with 50 ppm SO_2 have a lower propensity to sinter than those precalcined without SO_2. It must be assumed, therefore, that the 7.7% calcium conversion achieved during precalcination with 50 ppm SO_2 is sufficient to block much of the sulfation during reinjection. Sorbents precalcined with 10 ppm SO_2 appear to produce better calcium conversions during reinjection than sorbents precalcined without SO_2.

DISCUSSION

Contrary to the observations of Glasson[15] and Newton[16] the results presented in this paper suggest that the presence of 300 ppm or less SO_2 during calcination significantly reduces the subsequent propensity of the sorbent to sinter. While it would be desirable to reconcile these apparently conflicting observations, differences in the experimental frameworks make this difficult. Glasson introduced sulfate into reagent grade $CaCO_3$ by washing it in dilute sulfuric acid to produce partial dissolution, and

subsequently drying it. These modified sorbents were then calcined in an electric furnace at 800-1,000°C for 2-20 hr. There are no similarities between Glasson's experiments and this work, and reasons for the differences in conclusions cannot be determined.

Newton[16] calcined $CaCO_3$ at 980°C for 0.5 s with and without 180 ppm SO_2 present. The sorbent calcined without SO_2 had a mean pore diameter of 64 Å and virtually no pore volume above 100 Å. The sorbent calcined with SO_2 had a mean pore diameter of 155 Å and 80% of its pore volume above 100 Å. Using the distributed pore model, Newton concluded that the buildup of product layer would not produce the observed changes, and that they must be the result of enhanced sintering in the presence of SO_2. Lacking pore size distribution data from this work, the two studies cannot be directly compared.

The results reported here are fully consistent with those of Kirchgessner and Jozewicz[18] in which higher porosities were observed for sorbents calcined in the presence of SO_2 than for sorbents calcined in the absence of SO_2. The hypothesis set forth in that work suggests that the presence of $CaSO_4$ product on pore faces interferes with the exchange of migrating reactant species

and lattice vacancies at the pore/sorbent interface. This in turn would retard the solid state diffusion process or sintering. This hypothesis still appears to be reasonable.

The results of this work suggest that precalcining sorbents in the presence of small amounts of SO_2 does not have a practical application in high temperature sorbent injection processes. Although it has been demonstrated that sorbents precalcined with 10 ppm SO_2 are more reactive during reinjection than sorbents calcined in the absence of SO_2, neither the modified nor the unmodified precalcined sorbent is as reactive with SO_2 as is the raw $Ca(OH)_2$. Raw $Ca(OH)_2$ sorbents typically have produced calcium conversions greater than 20% when injected at 1000°C for 1.0 s with 3000 ppm SO_2.

CONCLUSIONS

Based on structural characteristics, $Ca(OH)_2$ sorbents precalcined to CaO in the presence of 300 ppm or less SO_2 retain greater amounts of surface area and porosity than sorbents precalcined in the absence of SO_2. It is believed that this behavior results from the ability of small amounts of $CaSO_4$ product on pore faces to interfere in the solid state diffusion

process of sintering. After quantification, it would seem reasonable to include the effect of SO_2 on sintering in the Silcox equation.

Sorbents precalcined with 10 ppm SO_2 are more reactive with SO_2 during reinjection into the reactor than sorbents precalcined in the absence of SO_2. Increasing SO_2 during precalcination to 50 ppm reverses these results. Since no precalcined sorbent has been shown to be more reactive with SO_2 than is raw $Ca(OH)_2$, this process is not believed to have a practical application.

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